

Note

Syntheses, characterization and crystal structures of
[Cu(PCHO)₂(2,2'-bipy)][BF₄], [Cu₂(PCHO)₄(4,4'-bipy)][BF₄]₂
and [Cu₂(PCHO)₄(bptz)][BF₄]₂ (PCHO = *o*-(diphenylphosphino)
benzaldehyde and bptz = 3,6-bis(2'-pyridyl)-1,2,4,5-tetrazine)

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Abstract

Treatment of [Cu(PCHO)₂(NCMe)][BF₄] (**1**) (PCHO = *o*-(diphenylphosphino)benzaldehyde) with 2,2'-bipyridine produces the complex [Cu(PCHO)₂(2,2'-bipy)][BF₄] (**4**), while the reactions of **1** with 4,4'-bipyridine and 3,6-bis(2'-pyridyl)-1,2,4,5-tetrazine (abbreviated as bptz) afford the dinuclear complexes [Cu₂(PCHO)₄(4,4'-bipy)][BF₄]₂ (**5**) and [Cu₂(PCHO)₄(bptz)][BF₄]₂ (**6**), respectively. The crystal structures of **4–6** have been determined by an X-ray diffraction study. The coordination about the Cu⁺ ion is trigonal for **5** and tetrahedral for **4** and **6**, and there is no bonding between the aldehyde groups and the Cu⁺ ion in these compounds.

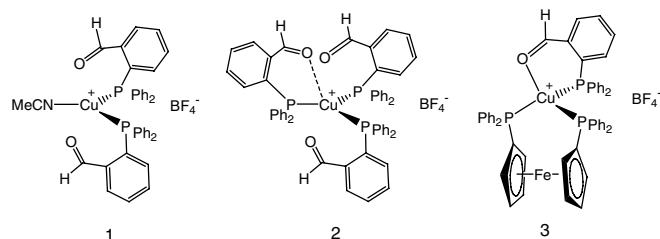
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Keywords: Cu(I) complexes; Phosphine ligands; Pyridine ligands

1. Introduction

The multifunctional, hemilabile ligands [1], which contain both soft (such as P, S) and hard (such as N, O) donor atoms, have attracted considerable interest for their unusual coordination chemistry and their increasing importance in catalysis [2]. The *o*-(diphenylphosphino)benzaldehyde molecule (abbreviated as PCHO) is one of the simplest bidentate P,O-donor agents [3]. Four modes of bonding have been observed in the interaction of PCHO with transition metal centers: η¹-P donor [4,5], η²-P,O chelating [6,7], η³-P,CO chelating [8], and P-acylhydride mode [9–14], which involves oxidative addition of the formyl C–H bond on the metals. Recently, we prepared the trigonal complex [Cu(PCHO)₂(NCMe)][BF₄] (**1**) [15] and found that replacement of the acetonitrile ligand by

PCHO afforded [Cu(PCHO)₃][BF₄] (**2**), showing a Cu–aldehyde interaction. In contrast, treating **1** with the bidentate phosphine (PPh₂C₅H₄)₂Fe (abbreviated as DPPF) led to dissociation of one PCHO ligand to produce the tetrahedral complex [Cu(PCHO)(DPPF)][BF₄] (**3**) containing an η²-P,O chelate. It was postulated that the steric properties of the neutral ligands play an important role in determining the cation stoichiometry [16]. To extend this scope, we present here the results concerning the substitution reactions of **1** with N-heterocyclic ligands.



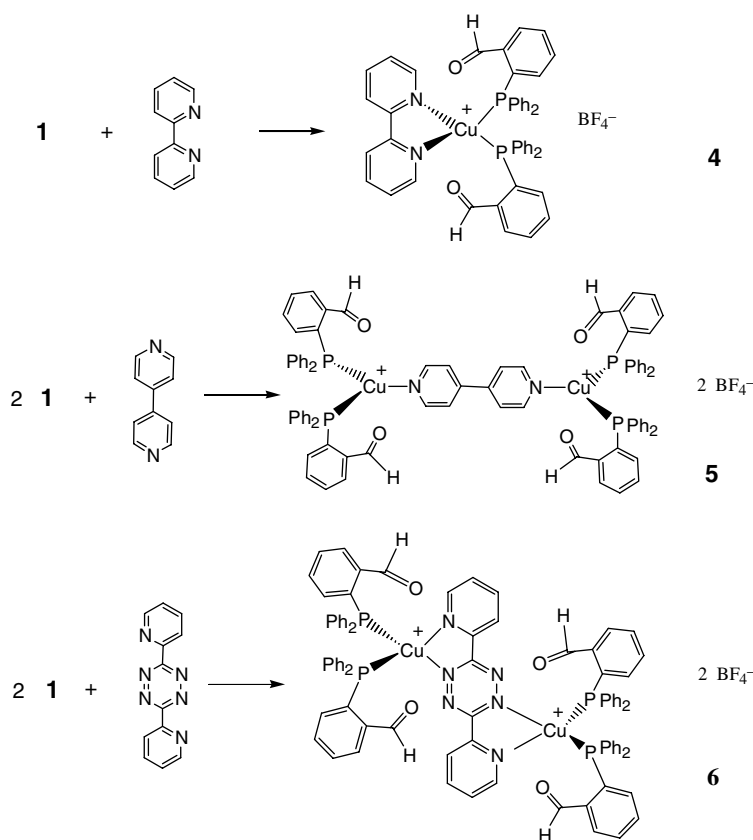
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2. Results and discussion

The reaction of **1** and 2,2'-bipyridine in THF solvent generated an orange precipitate, from which air-stable, orange-yellow crystals of the complex $[\text{Cu}(\text{PCHO})_2(2,2'\text{-bipy})][\text{BF}_4]$ (**4**) were obtained in 56% after recrystallization. On the other hand, treating **1** with 4,4'-bipyridine in a 2:1 molar ratio produced pale yellow crystals of the dinuclear complex $[\text{Cu}_2(\text{PCHO})_4(4,4'\text{-bipy})][\text{BF}_4]_2$ (**5**) in 63%. We then investigated the reaction of **1** with the multidentate ligand 3,6-bis(2'-pyridyl)-1,2,4,5-tetrazine (abbreviated as bptz), which took place in CH_2Cl_2 at ambient temperature to yield a brown solution, and the dinuclear complex $[\text{Cu}_2(\text{PCHO})_4(\text{bptz})][\text{BF}_4]_2$ (**6**; 42%) was obtained as air-stable, dark brown crystals after diffusion into diethyl ether. The reactions are summarized in Scheme 1. In contrast to compounds **2** and **3**, the formation of **4–6** does not alter the PCHO coordination. This difference is likely for steric reasons, since the planar N-heterocyclic ligands are less space-congested than the tertiary phosphines. Compound **5** is formally a 16-electron species, while **4** and **6** are 18-electron. They have been characterized by elemental analysis, mass, IR and NMR spectroscopy. The crystals suitable for an X-ray diffraction study were grown by diffusion of diethyl ether (for **4** and **5**) or *n*-hexane (for **6**) into a dichloromethane solution of the compound at ambient temperature.

The FAB spectrum of **4** displays the molecular cation at m/z 799. The ^1H NMR spectrum presents a 2H singlet at δ 9.51 for the pendant formyl protons, two 2H doublets at δ 8.84 ($J_{\text{H-H}} = 5$ Hz) and 8.31 ($J_{\text{H-H}} = 8$ Hz) and two 2H multiplets at δ 8.05 and 7.40 for the bipyridine protons, and a 28 H multiplet in the range δ 7.65–6.82 for the phenyl protons. In addition, the $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum shows a broad signal at δ 4.51 for the phosphine ligands. The spectral data are consistent with a C_2 symmetry for **4** in solution. The ORTEP diagram for the cation of **4** is depicted in Fig. 1. The coordination about the Cu^+ ion can be described as an irregular tetrahedron, where the angles around the copper atom range from $78.3(2)^\circ$ for N1-Cu-N2 to $126.92(6)^\circ$ for P1-Cu-P2 . The benzaldehyde rings are about parallel with a dihedral angle of 3.5° , and the mean separation of the planes is 3.60 Å. This value is compatible with 3.35 Å for the interplane distance in graphite and can be described in terms of a van der Waals type interaction [17]. The pyridine ligand chelates the Cu^+ ion with the N1-Cu length (2.110(5) Å) being slightly longer than the N2-Cu length (2.091(5) Å). The two pyridine rings are not coplanar, showing a dihedral angle of $8.6(6)^\circ$, and the Cu atom is 0.17(4) Å away from the mean plane of the N1, C6, C5 and N2 atoms. The $\text{O1}\cdots\text{Cu}$ and $\text{O2}\cdots\text{Cu}$ distances are 3.13 and 3.10 Å, respectively, too long to support a Cu–O interaction.



Scheme 1.

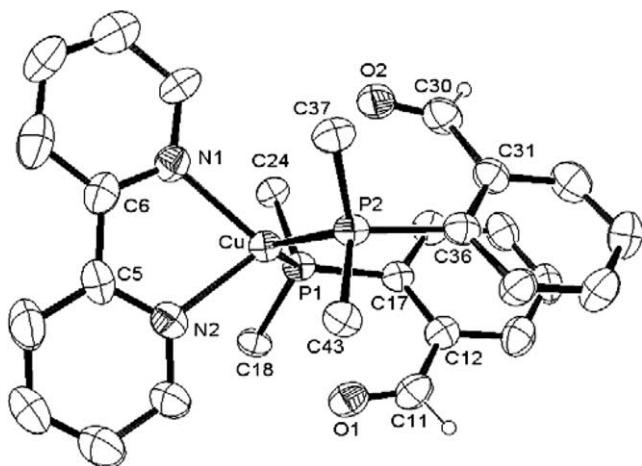


Fig. 1. Molecular structure of **4**. The BF_4^- anion has been artificially omitted. Only the *ipso* carbons of the C_6H_5 groups are shown for clarity. Selected bond distances (Å) and bond angles ($^\circ$): Cu–P1 2.269(2), Cu–P2 2.291(2), Cu–N1 2.110(5), Cu–N2 2.091(5), C11–O1 1.177(7), C30–O2 1.187(7), Cu \cdots O1 3.13, Cu \cdots O2 3.10 and P1–Cu–P2 126.92(6), N1–Cu–N2 78.3(2), P1–Cu–N1 113.5(1), P1–Cu–N2 111.0(2), P2–Cu–N1 107.3(1), P2–Cu–N2 109.3(1), Cu–P1–C17 117.1(2), Cu–P2–C36 117.8(2), C31–C30–O2 124.0(6), C12–C11–O1 125.4(6).

The FAB spectrum of **6** reveals an intense parent ion peak for the $[\text{Cu}_2(\text{PCHO})_4(\text{bptz})]^{2+}$ cation at m/z 761. The ^1H NMR spectrum at 25°C shows a broad signal at δ 9.62 for the free formyl protons and several multiplets in the range δ 9.23–6.90 for the aromatic protons. The ^{31}P resonance for the coordinated phosphine groups is recorded at δ 5.63. A thermal ellipsoid plot of the dinuclear cation $[\text{Cu}_2(\text{PCHO})_4(\text{bptz})]^{2+}$ is shown in Fig. 2. There is a crystallographic center of symmetry imposed on the molecule. The molecular cation consists of two $\text{Cu}(\text{PCHO})_2^+$ groups with each chelated by two nitrogen atoms of the bridging bptz ligand in a commonly encountered *anti* orientation [18], and the two Cu^+ ions are separated by

8.27 Å. The N3 atom is disordered at its site. Each Cu^+ ion adopts a distorted tetrahedral geometry similar to **4**, such that the N1–Cu–N2 and P1–Cu–P2 angles are $77.72(2)^\circ$ and $128.57(6)^\circ$, respectively. The mean Cu–N distance is 2.09 Å and Cu–P distance is 2.27 Å. The planar benzaldehyde groups are about parallel with a dihedral angle of 2.7° , and the mean separation of the planes is 3.58 Å. The O1 \cdots Cu and O2 \cdots Cu distances are 3.00 and 3.02 Å, respectively. We note that the geometry about the copper atom is similar to the related complex $[\text{Cu}_2(\text{PPh}_3)_4(\text{bptz})][\text{BF}_4]_2$ containing triphenylphosphine ligands [19].

Complex **5** exhibits the expected C_{2h} symmetry in solution. The ^1H NMR spectrum at 25°C shows a broad signal at δ 9.82 for the formyl protons, two doublet signals ($J_{\text{H-H}} = 6$ Hz) at δ 8.52 and 7.92 for the bipyridine protons, and a multiplet in the range δ 7.71–6.99 for the phenyl protons. The ^{31}P resonance is recorded at δ 1.77 as a broad signal. The structure of **5** consists of discrete cations $[\text{Cu}_2(\text{PCHO})_4(4,4'\text{-bipy})]^{2+}$ and tetrafluoroborate counter anions. There is a crystallographic center of symmetry imposed on the molecule. The ORTEP drawing for the cation is illustrated in Fig. 3. The complex contains two $\text{Cu}(\text{PCHO})_2^+$ moieties bridged by a 4,4'-bipyridine ligand, where the Cu \cdots Cu distance is 11.09 Å. With the N1, P1 and P2 atoms forming the basal trigonal plane, the interligand bond angles surrounding the Cu atom are within 5° of the trigonal expectation of 120° , and the three angles sum to 359.9° . The Cu atom is slightly displaced from the basal trigonal plane by 0.03 Å, which is within those measured for **1** (0.01 Å) and **2** (0.08 Å). The mean Cu–P distance of 2.24 Å is same as **1**, but is slightly shorter than the tetrahedral complexes **4** (2.28 Å) and **6** (2.27 Å). The pyridine ring is about perpendicular to the CuP_2 plane with a dihedral angle of 88° . Each benzaldehyde group is planar with the formyl oxygen pointing to the copper center. However,

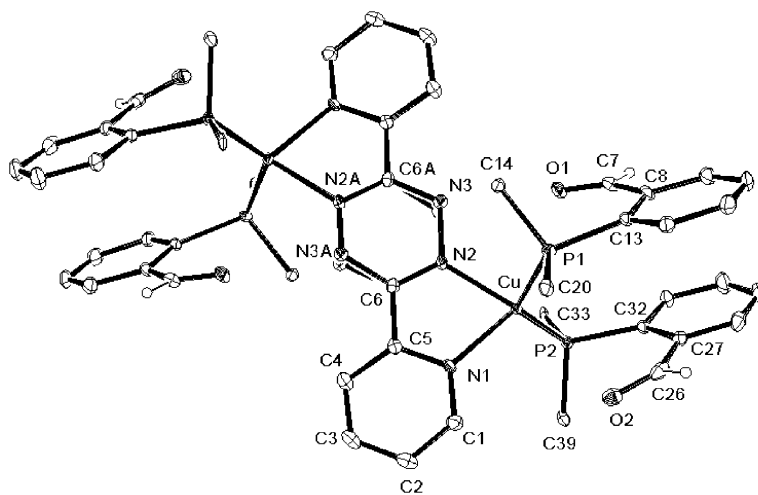


Fig. 2. Molecular structure of **6**. The BF_4^- anions have been artificially omitted. Only the *ipso* carbons of the C_6H_5 groups are shown for clarity. The N3 atom is disordered at its site. Selected bond distances (Å) and bond angles ($^\circ$): Cu–P1 2.281(2), Cu–P2 2.250(2), Cu–N1 2.087(4), Cu–N2 2.095(4), C7–O1 1.205(7), C26–O2 1.214(8), Cu \cdots O1 3.00, Cu \cdots O2 3.02 and P1–Cu–P2 128.57(6), N1–Cu–N2 77.7(2), P1–Cu–N1 112.6(1), P1–Cu–N2 110.3(1), P2–Cu–N1 111.0(1), P2–Cu–N2 114.6(1), Cu–P1–C13 119.5(2), Cu–P2–C32 114.5(2), C27–C26–O2 125.6(5), C8–C7–O1 124.6(6).

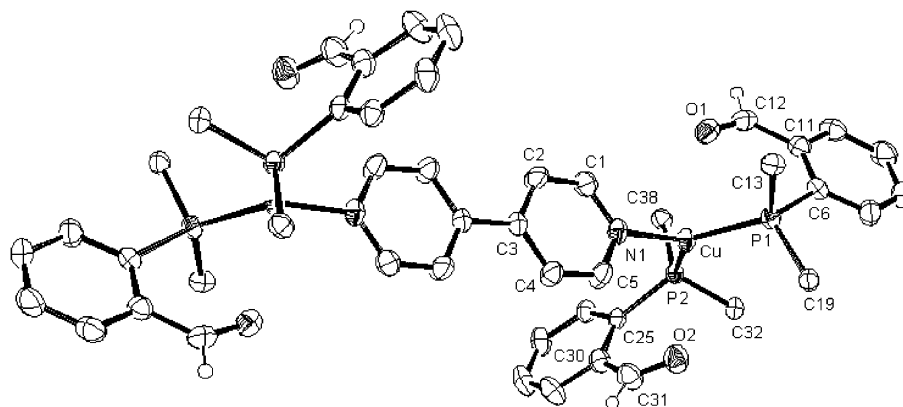


Fig. 3. Molecular structure of **5**. The BF_4^- anions have been artificially omitted. Only the *ipso* carbons of the C_6H_5 groups are shown for clarity. Selected bond distances (Å) and bond angles ($^\circ$): Cu–P1 2.2386(7), Cu–P2 2.2397(6), Cu–N1 1.996(2), C12–O1 1.207(3), C31–O2 1.218(3), Cu \cdots O1 2.67, Cu \cdots O2 2.83 and P1–Cu–P2 124.85(2), P1–Cu–N1 116.59(6), P2–Cu–N1 118.50(6), Cu–N1–C1 124.0(2), Cu–N1–C5 119.6(2), Cu–P1–C6 111.69(8), Cu–P2–C25 117.84(7), C30–C31–O2 124.6(3), C11–C12–O1 125.8(2).

the two benzaldehyde rings are not in close proximity, while they are parallel and have a van der Waals type interaction in **1**, **2**, **4** and **6**. Such difference is reflected to the O1 \cdots Cu \cdots O2 angle, which is 115.7° for **1**, 108.8° for **2**, 113.8° for **4** and 114.9° for **6**, but is 169.7° for **5**. The O1 \cdots Cu distance (2.67 Å) and O2 \cdots Cu distance (2.83 Å) are much shorter than those in **4** and **6** and could suggest a certain extent of dipole–ion interaction [20].

In conclusion, our studies have provided a useful range of structural data for $[\text{Cu}(\text{PCHO})_2\text{X}]$ complexes, in which X presents P- or N-donor ligands with different steric profiles. It is promising that, by selective oxidation of the Cu $^+$ ions, the dinuclear complexes **5** and **6** might provide simple models for the study of the magnetic interaction of unpaired *d* electrons [21]. The investigation is progressive in our laboratory.

3. Experimental

3.1. General methods

All manipulations were carried out under an atmosphere of dinitrogen with standard Schlenk techniques. Compound **1** [15] and bptz [22] were prepared by the literature methods. 2,2'-Bipyridine and 4,4'-bipyridine were purchased from Aldrich and used as received. The solvents were dried over appropriate reagents under dinitrogen and distilled immediately before use. ^1H and ^{31}P NMR spectra were obtained on a Varian Unity INOVA-500 spectrometer. Fast-atom-bombardment (FAB) mass spectra were recorded on a JEOL JMS-SX102A mass spectrometer. Elemental analyses were performed at the National Science Council Regional Instrumentation Center at National Chen-Kung University, Tainan, Taiwan.

3.2. Preparation of **4**

Compound **1** (22 mg, 0.03 mmol), 2,2'-bipyridine (7 mg, 0.045 mmol) and THF (4 ml) were placed in an oven-dried

20 ml Schlenk tube. The solution was gently heated to reflux for 40 min and then cooled to ambient temperature to produce an orange precipitate. The mixture was filtered, and the solid was extracted with dichloromethane. The dichloromethane solution was carefully layered with diethyl ether to afford $[\text{Cu}(\text{PCHO})_2(2,2'\text{-bipy})][\text{BF}_4]$ (**4**) (15 mg, 56%) as an air-stable, orange-yellow crystalline solid. *Anal.* Calc. for $\text{C}_{48}\text{H}_{38}\text{BCuF}_4\text{N}_2\text{O}_2\text{P}_2$: C, 64.99; H, 4.32; N, 3.16. Found: C, 65.25; H, 4.30; N, 3.01%. Mass (FAB): m/z 799 ($[\text{Cu}(\text{PCHO})_2(2,2'\text{-bipy})]^+$; ^{63}Cu). ^1H NMR (CD_2Cl_2 , 25 $^\circ\text{C}$): δ 9.51 (s, 2H, CHO), 8.84 (d, 2H, $J_{\text{H-H}} = 5$ Hz, bipy), 8.31 (d, 2H, $J_{\text{H-H}} = 8$ Hz, bipy), 8.05 (m, 2H, bipy), 7.40 (m, 2H, bipy), 7.65–6.82 (m, 28H, Ph). $^{31}\text{P}\{^1\text{H}\}$ NMR (CD_2Cl_2 , 25 $^\circ\text{C}$): δ 4.51 (br). IR (CH_2Cl_2 , cm^{-1}): 1690 (C=O).

3.3. Preparation of **5**

Compound **1** (45 mg, 0.06 mmol), 4,4'-bipyridine (5 mg, 0.03 mmol) and dichloromethane (3 ml) were placed in an oven-dried 20 ml Schlenk tube. The solution was stirred at ambient temperature for 8 h and carefully topped with diethyl ether (10 ml). $[\text{Cu}_2(\text{PCHO})_4(4,4'\text{-bipy})][\text{BF}_4]_2$ (**5**) (41 mg, 63%) was obtained as air-stable, pale yellow crystals. *Anal.* Calc. for $\text{C}_{86}\text{H}_{68}\text{B}_2\text{Cu}_2\text{F}_8\text{N}_2\text{O}_4\text{P}_4$: C, 63.84; H, 4.24; N, 1.73. Found: C, 64.05; H, 4.27; N, 1.68%. Mass (FAB): m/z 643 ($[\text{Cu}(\text{PCHO})_2]^+$; ^{63}Cu). ^1H NMR (CDCl_3 , 25 $^\circ\text{C}$): δ 9.82 (br, 4H, CHO), 8.52 (d, 4H, $J_{\text{H-H}} = 6$ Hz, bipy), 7.92 (d, 4H, bipy), 7.71–6.99 (m, Ph). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , 25 $^\circ\text{C}$): δ 1.77 (s). IR (CH_2Cl_2 , cm^{-1}): 1674 (C=O).

3.4. Preparation of **6**

Compound **1** (45 mg, 0.061 mmol) and bptz (7.2 mg, 0.031 mmol) were placed in a Schlenk flask under dinitrogen, and dichloromethane (5 ml) was introduced. The resulting mixture was stirred at ambient temperature for 3 h, yielding a dark brown solution. The solution was

Table 1
Crystallographic data for 4–6

	4	5	6
Formula	C _{48.75} H _{39.5} BCl _{1.5} CuF ₄ N ₂ O ₂ P ₂	C ₈₆ H ₆₈ B ₂ Cu ₂ F ₈ N ₂ O ₄ P ₄	C ₉₁ H ₇₄ B ₂ Cl ₆ Cu ₂ F ₈ N ₆ O ₄ P ₄
Crystal system	monoclinic	monoclinic	triclinic
Formula weight	950.79	1618.00	1952.84
T (K)	295	150	150
Space group	P2 ₁ /n	P2 ₁ /c	P $\bar{1}$
a (Å)	9.8664(2)	10.5686(4)	13.072(1)
b (Å)	25.4346(4)	17.9957(7)	13.763(1)
c (Å)	19.9104(3)	20.6877(8)	14.896(1)
α (°)	90	90	109.170(2)
β (°)	100.9735(9)	99.936(1)	90.921(2)
γ (°)	90	90	117.925(2)
V (Å ³)	4905.1(2)	3875.6(3)	2189.9(3)
Z	4	2	1
D _{calc} (Mg/m ³)	1.287	1.387	1.481
μ (mm ⁻¹)	0.646	0.703	0.814
Final R indices (I > 2 σ (I))	0.0833/0.2487	0.0463/0.1110	0.0815/0.1838
Goodness-of-fit on F ²	1.023	1.035	1.210

diffused into diethyl ether (15 ml) to afford air-stable, brown crystals of [Cu₂(PCHO)₄(bptz)]₂[BF₄]₂ (**6**) (22 mg, 42%). *Anal. Calc.* for C₈₈H₆₈B₂Cu₂F₈N₆O₄P₄: C, 62.24; H, 4.04; N, 4.95. Found: C, 63.02; H, 3.98; N, 4.90%. Mass (FAB): *m/z* 761 (M²⁺, ⁶³Cu). ¹H NMR (CD₂Cl₂, 25 °C): δ 9.62 (br, CHO), 9.23–6.90 (m, Ph and bptz). ³¹P{¹H} NMR (CD₂Cl₂, 25 °C): δ 5.63 (br). IR (CH₂Cl₂, cm⁻¹): 1688 (C=O).

3.5. Structure determination for 4–6

The crystals of 4–6 found suitable for X-ray analysis were each mounted in a thin-walled glass capillary and aligned on the Nonius Kappa CCD (for 4) and Bruker Smart Apex CCD (for 5 and 6) diffractometers, with graphite-monochromated Mo K α radiation ($\lambda = 0.71073$ Å). The θ range for data collection is 1.31–25.00° for 4, 1.51–27.50° for 5 and 1.48–25.00° for 6. Of the 39861, 37546 and 17762 reflections collected for 4, 5 and 6, 8642, 8896 and 7732 reflections were independent, respectively. All data were corrected for Lorentz and polarization effects and for the effects of absorption. The structure was solved by the direct method and refined by least-square cycles. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. All calculations were performed using the SHELXTL-97 package [23]. The data collection and refinement parameters are listed in Table 1.

4. Supplementary material

Crystallographic data for 4–6 have been deposited with the Cambridge Crystallographic Data Centre, Deposition Nos. CCDC 268482 for 4, 249803 for 5 and 268481 for 6. Copies of these data may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk or on the web www: <http://www.cam.ac.uk>).

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